IS 8642 (1977): Dyes for water based writing inks [CHD 14: Printing, Inks, Stationary and Allied Products]

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IS : 8642 - 1977
(Superseding IS : 2247 - 1962)
(Reaffirmed 2010)
Edition 1.1
(1986-01)

Indian Standard

SPECIFICATION FOR DYES FOR WATER-BASED WRITING INKS

(Incorporating Amendment No 1)

UDC 667 271 667 4

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Price Group 6

Plastres Containers Sectional Committee, PCD 21

FOREWORD

This Indian Standard (Second Rcvision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Plastres Containers Sectional Committee had been approved by the Petroleum. Coal and Related Products Division Council

This standdard was originally published in 1977 and revised in 1988 The present revision has been taken up with a view to include test for overall migration with water and requirements of mass and dimensions List of plastics materials which have been recommended as suitable fof packing of mineral water in IS 10171 19K7 'Guide on suitability ol plastus for food packaging' has been included

A separate specification exists for plastic packaging of natural mineral water and packaged drinking water

A scheme of labelling environment frientlly products with the FCO logo has been introduced at the instance ol the Ministry of Environmeny and Eorests (MI 1), Government of India the LCO Mark is being administered by the Bureau of Indian Standards (BIS) under the *BIS Act*, 1986 as per the Resolutions No 71 daled 21 February 1991 and No 425 dated 28 October 1992 published in the Gazette of the Government of India For a product to he eligible for marking with the ECO logo, it shall also carry the ISI Mark of the BIS besides meeting additional environment friendly requirements. For this purpose the Standard Mark would be a single mark being a combination of the ISI Mark and the I C O logo

These requirements are based on the gazette Notification No 170 dated 18 May 1996 for plastic products as environment friendly products.s published in the Gazette of the Government of India

In reprinting the results of a test on analysis made in accordance with this standard, of the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 1960 'Rules for rounding off numerical values (revised) The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard

IS : 8642 - 1977

(Superseding IS : 2247 - 1962)

Indian Standard

SPECIFICATION FOR DYES FOR WATER-BASED WRITING INKS

Inks and Allied Products Sectional Committee, CDC 13

Chairman

DR M I MATHEWS

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Indian Standard

SPECIFICATION FOR DYES FOR WATER-BASED WRITING INKS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 14 October 1977, after the draft finalized by the Inks and Allied Products Sectional Committee had been approved by the Chemical Division Council

0.2 The quality of writing ink will, among other factors, depend upon the quality of the dye used in the ink The manufacturers of inks had been experiencing considerable difficulty in obtaining the requisite quality of dye from indigenous sources. It was, therefore, considered important to formulate a standard for dyes used in ink industry It is expected that this standard will help the producers of ink to procure the dyes of assured quality in the country

0.3 In the preparation of this standard considerable assistance has been derived from the investigations carried out by Atul Products Ltd, Bulsar and Kores (India) Ltd, Bombay and the assistance so derived is thankfully acknowledged.

0.4 This standard covers the requirements of the various dyes used in writing inks including that of ink blue currently covered under IS - 2247-1962* which will be withdrawn on the publication of this standard

The dyes covered under this standard are ink blue, acid green, acid violet, nigrosine, direct black, crocein scarlet, eosine, acid fast scarlet, tartrazine yellow and acid black

0.5 This edition 1 1 incorporates Amendment No. 1 (January 1986), Side bar indicates modification of the text as the result of incorporation of the amendment

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS · 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Specification for dye, ink blue, for ink industry

†Rules for rounding off numerical values (*revised*)

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for dyes used in the manufacture of water-based writing inks.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in IS · 4395-1967*, in addition to the following shall apply

2.1.1 *Approved/Standard Sample* — The sample accepted by the purchaser or inspection authority as the basis of supply or manufacture.

3. REQUIREMENTS

3.1 Description — The dyes have hue number, colour index number†, chemical name, molecular mass and equivalent mass as given in Table 1

3.2 Identification — The material shall comply with the identification requirements given in Table 2 when tested by the methods prescribed in Appendix A Reference to the relevant clauses of Appendix A is given in col 13 of the table

3.3 The material shall also comply with the requirements laid down in Table 3 when tested by the methods prescribed in Appendix A Reference to the relevant clauses of Appendix A is given in col 13 of the table.

3.4 Presence of Starch, Dextrin and Gum — The material shall pass the test prescribed in **A-14.**

4. PACKING AND MARKING

4.1 Packing — Unless otherwise agreed between the purchaser and the supplier the material shall be packed in sound, clean and dry tinplate containers fitted with lever lids

4.2 Marking — The containers shall be legibly marked with the following information

a) Description of the material;

b) Tare and net mass;

c) Manufacturer's name and/or recognized trade-mark, if any, and

d) Batch number in code or otherwise to enable the lot and date of manufacture to be traced from records.

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*Glossary of terms relating to inks and allied industries

†Colour Index, 1956, Second Edition Published by Society of Dyers and Colourists. U K. and American Association of Textile Chemists and Colourists.

4.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inapection, testing and quahty control which is devised and supervised by ISI and operated by the producer ISI marked products are also contmuously chocked by ISI for conformity to that slandard as a further safeguard Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution

5. SAMPLING

5.1 The method of drawing representative samples of the material, number of tests to be performed and the criteria for judging the conformity of the material to the requirements of this specification shall be as prescribed in Appendix B.

TABLE 1 DESCRIPTION OF DYES FOR WATER-BASED WRITING INKS

(*Clause* 3 1)

SL No	NAME OF THE DYE	HUE NO	Colour INDEX No	CHEMICAL NAME	MOLECULAR MASS	EQUIVALENT MASS
(1)	(2)	(3)	(4)	(5)	(6)	(7)
1)	Ink blue	Acid Blue 93	42780	Ammonium salt of triphenyl triamino triphenylcarbinol trisulphonic acid	—	—
ii)	Acid green	Acid green 16	444025	4-4' dimethylamino-phenyl-methane naphthyl-3.6' disodium sulphonate	684 (as acid)	
iii)	Acid violet	Acid Violet 17	42650	N. N-ethyl benzoyl aniline formaldehyde, N N diethyl aniline	740 (as acid)	385
iv)	Nigrosine	Acid black 2	50420	Sodium salt of sulphonated nigrosine base which is a camplex mixture of phenylated indulines of unspecified structure	—	—
V)	Direct black	Direct black	30235	Sodium salt of benzene azo-3 6 disulfo-8-amino-1-naphtol-7-azo-diphenylazo-m-phenylene diamine	787 74 (as acid)	61 47
vi)	Crocein scarlet M00	Acid red 73	27290	Sodium salt of benzene azo-benzene azo-2-naphthol-6 8 disulfonic acid	512 4 (as acid)	64 06
vii)	Eosine	Acid red 87	45380	Disodium salt of tetrabromo-fluoreseein	691 9	—
viii)	Acid fast scarlet	Acid red 18	16255	Trisodium salt of 1-(4-sulfo-I-naphtyiylazo)-2-hydroxy napthalene 6 8 disulfonic acid	604 5	151 1
ix)	Tartrazine yellow	Acid yellow 23	19140	Trisodium salt of 4-(4'-sulfo-l-phenylazo)-l-(4'-bulpho-phenyl)-5-hydroxy pyrazol -3- carboxylic acid	534 4	133 6
x)	Acid black	Acid black 1	20470	Sodium salt of 1-nitrobenzene azo-3 6-disulfo-l-amine-8-naphthol azo benzene	572	40 89

TABLE 2 IDENTIFICATION REQUIREMENTS OF DYES FOR WATER-BASED WRITING INKS

(*Clause* 3 2)

SL No.	CHARACTERISTIC	IDENTIFICATION REQUIREMENTS										METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
		Ink Blue	Acid Green	Acid Violet	Nigrosine	Direct Black	Crocein Scarlet M00	Eosine	Acid Fast Scarlet	Tartrazine Yellow	Acid Black	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)
i)	Physical appearance	Lumps, granules or powder with deep brown golden lustre	Brownish black powder	Brownish — violet powder with lustre		Greyish black powder	Scarlet powder	Scarlet powder	Dark scarlet powder	Orange powder	Dark brown powder	—
ii)	Colour of acqueous solution (0 5 percent) *mlv*	Blue	Bluish green	Violet		Greenish black	Red	Bluish red with a yello wish green fluorescence	Deep red	Yellow	Black	—
iii)	Action of acid											A-2
	a) Dilute hydrochloric acid	Blue solution	Ye11owish brown solution	Dark green — solution		Blackish solution with precipitate	Blackish brown solution With precipitate	Orange solution with precipitate	Deep red solution	Yellow solution	Black solution with precipitate	
	b) Concentrated hydrochloric acid	Blue solution	Brownish orange solution	Reddish — brown solution		Dull reddish black solution with precipitate	Blackish brown solution with precipitate	Orange solution with precipitate	Deep red solution	Yellow solution	Bluish black solution with precipitate	
	c) Dilute sulphuric acid	Blue solution	Yellowish brown solution	Violet solution —		Reddish black solution with precipitate	Reddish brown solution with precipitate	Ordnge solution with precipitate	Reddish orange solution	Yellow solution	Greenish black solution with precipitate	
	d) Concentrated sulphuric acid	Reddish brown	Ye11owish brown solution	Reddish — yellow solution		Bluish black solution with precipitate	Reddish violet solution with precipitate	Yellow solution	Violet solution	Yellow solution	Deep green solution with precipitate	
iv)	Action of Alkali											A-3
	a) Sodium hydroxide solution	Reddish brown solution	Bluish green solution	Bluish violet —		Greyish blue solution with precipitate	Reddish brown solution with precipitate	Reddish	Reddish brown solution	Reddish yellow solution	Greenish solution	
	b) Sodium carbonate sotution	Violet solution	Bluish green solution	Bluish violet solution	—	Bluish grey solution	Red solution	Red solution	Deep red solution	Yellow solution	Bluish black solution	

TABLE 3 REQUIREMENTS OF DYES FOR WATER-BASED WRITING INKS

(*Clause* 3 3)

SL No	CHARACTERISTIC	REQUIREMENT FOR										METHOD OF TEST (REF TO CL NO IN APPENDIX A)
		Ink Blue	Acid Green	Acid Violet	Nigrotsine	Direct Black	Crocein Scarlet M00	Eosine	Acid Fast Scarlet	Tartrazine Yellow	Acid Black	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)
i)	Dye content, percent by mass	*	*	*	*	57 ± 2	62 ± 2	—	80 ± 2	80 ± 2	62 ± 2	A-4
ii)	Strength, percent											
	a) Byspectrophotometeror colorimeter	100 ± 2	100 ± 2	100 ± 2	100 ± 2	100 ± 2	100 ± 2	100 ± 2	100 ± 2	100 ± 2	—	A-5
	b) By dyeing	← Close match with the approved/standard sample →										A-6
iii)	Volatile matter, percent by mass *Max*	10 0	5 0	5 0	—	10 0	5 0	10 0	5 0	10 0	10 0	A-7
iv)	*p*H value (of aqueous solution)	4 0 to 4 5	8 0 ± 0 5	8 0 ± 0 5	—	10 0 ± 0 5	10 0 ± 0 5	10 0 ± 0 5	8 0 ± 0 1	8 0 ± 0 5	10 0 ± 0 5	A-8
v)	Matter insoluble in water percent by mass, *Max*	0 2	1 0	1 0	—	1 0	1 0	1 0	1 0	1 0	1 0	A-9
vi)	Colour value (of 0 002 5 percent aqueous, solution)	0 9R + 2 0B	4 0Y + 0 3R + 9 0B	13 5R + 7 1 B	—	0 7Y + 3 5R + 3 0B	2 0Y + 7 7R	10 8Y + 23 9 R‡	0 8Y + 7R‡	23Y + 3 7R + 0 3B	4 0Y + 2 9R + 3 0B	A 10
vii)	Shade	← Close match with the approved/standard sample →										A-11
viii)	Calcium oxide (as CaO), percent by mass, *Max*	0 8	—	—	6 0	—	—	—	—	—	—	A 12
ix)	Sulphated ash percent by mass, *Max*	5 0	—	—	—	—	—	—	—	—	—	A-13

*The determination oi strength by reduction with titanium trichloride is not posible as these dyes do not belong to azo group of dyes

‡The values are for 0 5 percent solution

APPENDIX A

(*Clauses* 3.2, *and* 3 3 and *Tables* 2 *and* 3)

METHODS OF TEST FOR DYES FOR WATER-BASED WRITING INKS

A.I. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS . 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals shall mean chemicals that do not contain impurities which affect the results of analysis

A-2. TEST FOR ACTION OF ACID

A-2.1 Reagents

A-2.1.1 *Dilute Hydrochloric Acid* — 10 percent (*v/v*)

A-2.1.2 *Concentrated Hydrochloric Acid — See* IS . 265-1962†.

A-2.1.3 *Dilute Sulphuric Acid* — 10 percent (*v/v*)

A-2.1.4 *Concentrated Sulphuric Acid — See* IS 266-1961‡

A-2.2 Procedure — Take 0.5 g of the dye and dissolve in 100 ml of water Transfer 5 ml of this solution to a test tube and add 5 ml of dilute hydrochloric acid. Observe the colour of the solution and the precipitate, if any Carry out similar tests with concentrated hydrochloric acid, dilute sulphuric acid and concentrated sulphuric acid.

A-2.2.1 The material shall satisfy the requirement of the test if the action produced is the same as specified in Table 2

A-3. TEST FOR ACTION OF ALKALI

A-3.1 Reagents

A-3.1.1 *Sodium Hydroxide Solution* — 10 percent (m*/v*)

A-3.1.2 *Sodium Carbonate Solution* — 10 percent (*m/v*).

A-3.2 Procedure — Take 0.5 g of the dye and dissolve it in 100 ml of water. Transfer 5 ml of this solution to a test tube and add 5 ml of sodium hydroxide solution Observe the colour of the solution and the precipitate, if any. Carry out similar tests with sodium carbonate solution

*Specification for water for general laboratory use (*second revision*)
†Specification for hydrochloric acid (*revised*)
‡Spacification for sulphuric acid (*revised*).

A-3.2.1 The material shall satisfy the requirement of the test if the action produced is the same as specified in Table 2.

A-4. DETERMINATION OF DYE CONTENT

A-4.1 Reagents

A-4.1.1 *Ammonium Thiocyanate Solution* — 20 percent (*m/v*).

A-4.1.2 *Dilate Hydrochloric Acid* — 25 percent (*m/v*)

A-4.1.3 *Titanium Trichloride Solution* — approximately 0 025 N, prepared as follows

Prepare a 15 percent (*m/v*) solution of titanium trichloride. Take 30 ml of the solution. Filter it through a thick pad of glass wool. Add this solution to a previously boiled mixture of 1 litre of water and 60 ml of concentrated hydrochloric acid Mix this solution by passing into it a current of inert gas, such as carbon dioxide or nitrogen, for about 15 minutes. Store the mixture in a bottle in an atmosphere of carbon dioxide.

NOTE — Carbon dioxide may be supplied by Kipp's apparatus The solution shall be stored in a bottle painted black to protect it from light

A-4.1.4 *Ferric Ammonium Sulphate Solution* — saturated solution (see IS. 2263-1962*).

A-4.2 Procedure

A-4.2.1 Weigh accurately about 1 g of the dye and dissolve it in hot water. Pour the solution into a 500-ml graduated flask and make the volume with water to 500-ml mark Mix well

A-4.2.2 Pipette out 50 ml of the solution into a 500-ml reduction flask containing 50 ml of dilute hydrochloric acid and 50 ml water which have been previously brought to boil. Start passing carbon dioxide into the solution and simultaneously heat the contents to boil.

A-4.2.3 Pass carbon dioxide in the storage bottle containing titamuin trichloride solution and immediately draw 50 ml of titanium trichloride solution by a pipette in which carbon dioxide has been passed previously and add this solution to the dye solution (*see* **A-4.2.2**). Immediately boil the contents for 10 to 15 minutes for complete reduction. Cool the flask in an ice-bath. Maintain continuous flow of carbon dioxide throughout the test. Add 10 ml of ammonium thiocyanate indicator solution Titrate the mixture against ferric ammonium sulphate solution to a permanent change of colour. Note

*Methods of preparation of indicator solutions for volumetric analysis

the volume of ferric ammonium sulphate solution (*see* Note).

NOTE — The volume of ferre ammonium sulphate solution required for titration should not he less than 10 ml

A-4.2.4 Carry out a blank by following the same procedure and the same quantities of the reagents but using 50 ml of water in place of the dye solution

A-4.3 Calculation

$$\text{Dye content, percent by mass} = \frac{(B-A)\times N\times D\times V}{H\times C\times M\times 10}$$

where

B = volume in ml of ferre ammonium sulphate solution required for the blank (*see* **A-4.2.4**),

A = volume in ml of ferric ammonium sulphate solution required for the dye,

N = normality of ferric ammonium sulphate solution,

D = molecular mass of the dye,

V = volume of dye solution (*see* **A-4.2.1**),

H = number of hydrogen atoms consumed per molecule of the dye,

C = volume in ml of aliquot taken for titration (*see* **A-4.2.2**), and

M = mass in g of the sample taken for the test

A-4.4 Carry out the test for approved/standard sample also under identical conditions using standard dye in place of sample dye

NOTE — Each azo group requires four mitro group, six hydroze group and two atoms of hydrogen for reduction.

A-5. DETERMINATION OF STRENGTH BY SPECTROPHOTOMETER OR COLORIMETER

A-5.1 By Spectrophotometer Method

A-5.1.1 *Reagents*

A-5.1.1.1 *Buffer solution* — of approximate *p*H 5 for ink blue, acid green, acid violet nigrosine (as acid black), crocein scarlet and eosine (as acid red), and 9 for direct black.

A-5.1.2 *Procedure*

A-5.1.2.1 Weigh accurately 1.0 g of the dye and dissolve it in hot water Pour the solution into 1-litre graduated flask and make up the volume with water up to the mark. Mix well

A-6.1.2.2 Pipette out 10 ml aliquot from the dye solution and pour it in 1-litre graduated flask. Add 50 ml of the buffer solution and finally make up the volume with water to 1 litre mark Mix well.

A-5.1.2.3 Prepare solution of the approved/standard samples as described in **A-5.1.2.1** and **A-5.1.2.2.**

A-5.1.2.4 Prepare two absorption 1-cm cells for use which are satisfactorily matched to each other in light transmittance when filled with water. In one cell, take water as a reference solution and in another cell, take the dye solution of the sample as prepared in **A-5.1.2.2.** Measure the optical density of the solution at the following wavelengths

Ink blue	550 nm
Acid green	605 nm
Acid violet	530 nm
Nigrosine as acid black	618 nm
Direct black	484 nm
Crocem scarlet	510 nm
Eosine as acid red	510 nm

A-5.1.2.5 Similarly take the optical density of the approved/standard dye solution prepared in **A-5.1.2.3** following the same procedure as in **A-5.1.2.4.**

A-5.1.3 *Calculation* — Calculate the percentage atrength of the dye sample considering the optical density of the approved/standard sample as 100 percent

$$\text{Strength of dye, percent} = \frac{D_1}{D_2} \times 100$$

where

D_1 = optical density of the sample, and

D_2 = optical density of the approved/standard dye.

A-5.2 By Photoelectric Colorimeter

A-5.2.1 *Reagent*

A-5.2.1.1 *Buffer solution* — Same as **A-5.1.1.1.**

A-5.2.2 *Procedure* — Weigh accurately 1 000 g of the sample and dissolve it in hot water. Pour the solution into 1-litre volumetric flask and make up the volume with water up to the mark. *Mix.* well. Pipette out 10 ml aliquot of the solution and transfer it to another 1-litre graduated flask. Add 50 ml of the buffer solution and finally make up the volume with water up to 1-htre mark Mix well. Prepare the

solution of the approved/standard dye in a similar manner. Measure the percentage transmission of the sample solution on a photoelectric calorimeter using suitable filters. Similarly measure the percentage transmission of the approved/standard dye solution

A-5.2.3 *Calculations* — Calculate the percentage strength of the dye considering the strength of the approved/standard solution as 100 percent

$$\text{Strength of the dye, percent} = \frac{T_1}{T_2} \times 100$$

where

T_1 = percentage transmission of the sample, and
T_2 = percentage transmission of the approved/standard dye.

A-6. DETERMINATION OF STRENGTH BY DYEING

A-6.1 Reagents

A-6.1.1 *Sodium Sulphate (Calcined) Solution* — 10 percent (*mlv*)

A-6.1.2 *Sodium Chloride Solution* — 10 percent (m/*v*)

A-6.1.3 *Formic Acid* — 10 percent (*m/v*)

A-6.1.4 *Soda Ash Solution* — 10 percent (*m/v*).

A-6.2 For Acid Dyes

A-6.2.1 *Preparation of Hanks for Dyeing*

A-6.2.1.1 Hanks of scoured 100 percent pure woollen knitting yarn (except for direct black in which case cotton yarn shall be used, see **A-6.3**) having no finishing chemicals, blueing or fluorescent brightening agents, shall be used. Each hank shall weigh 5 ± 0 1 g.

A-6.2.1.2 *Pretreatment of hanks* — The hank shall be heated in warm water (60°C) for 10 minutes, squeezed evenly to contain approximately its own mass of water, cooled and entered into the dye-bath.

A-6.2.2 *Preparation of Dyestuff Solution*

A-6.2.2.1 Weigh accurately 1 0 g of the dye Paste it thoroughly with cold water. Add hot water and dissolve the dye If necessary, heat the solution till it becomes clear. Dilute the solution to 1 litre with cold water.

A-6.2.2.2 Similarly prepare solution of the approved/standard dye following the procedure mentioned in **A-6.2.2.1**.

A-6.2.2.3 *Depth of shade* — Use medium shades (about 1.0 percent) as they show variation in strength more clearly than dark shades.

A-6.2.3 *Dyeing Procedure* — Pipette out 50 ml of the dye sample solution prepared as in **A-6.2.2.1**. Similarly pipette out separately 50 ml the approved/standard dye solution in other dye vessel Add 5 ml of sodium sulphate solution, 1 ml of formic acid and 250 ml of water in each dye vessel. Stir the dye liquor Place them in water-bath and raise the temperature to 50°C Introduce wetted hanks in each dye vessel and turn them frequently so as to obtain level dyeing. Work for 10 minutes at this temperature Remove the hanks from the dye vessels, add 1 ml of formic acid in each vessel and star well. Introduce the hanks into the dye-bath and turn the hanks frequently Slowly raise the temperature of dye-bath to boiling within 20 minutes Continue the dyeing at boil for 45 minutes. At the end of dyeing squeeze the dyed hank and rinse with warm water (60°C). Dry the hanks in an oven or in a drier at temperature not exceeding 70°C.

A-6.3 For Direct Black

A-6.3.1 *Preparation of Hanks for Dyeing*

A-6.3.1.1 A sufficient number of hanks of scoured, bleached, unmercensed cotton yarn having no finishing chemicals, blueing or fluorescent brightening agents, shall be used in this test. Each hank shall weigh 10 ± 0.1 g (*see* Notes below).

NOTE I — Anv yarn normally used in the laboratories for carrying out trials or yarn of the following requirements is suitable for this test

a) *Count* — 2/40s or 2/60S,

b) *Tuist per metre* — 750. and

c) *Cuprummomum fluidity* — not more than 5

NOTE 2 — If the mass of hank is not 10 ± 0 1 g then it should be weighed accurately and the amount of dyes and the chemicals to be taken should be calculated accordingly

A-6.3.1.2 *Pretreatment of test hanks* — The hank shall be treated in boiling water for 10 minutes, squeezed evenly to contain approximately its own mass of water, cooled and entered into the dye-bath.

A-6.3.2 *Preparation of Dyestuff solutions*

A-6.3.2.1 Weigh accurately l0g of the sample. Paste it thoroughly with cold water. Add hot water and dissolve the dye If necessary, heat the solution till it becomes clear. Dilute the solution to 1 litre with cold water

A-6.3.2.2 Similarly, prepare solution of the standard dye by following the procedure mentioned in **A-6.3.2.1** but taking the standard dye instead of the sample dyes.

A-6.3.3 *Dyeing Procedure* — Pipette out separately 100 ml of solution of the dye sample prepared as in **A-6.3.2.1** in the dye vessel so as to get one

percent depth (*see* **A-6.2.2.3**) Similarly, pipette out separately 100 ml of the solution of approved/standard dye in the other dye vessel Add 190 ml of water and 10 ml of sodium chloride solution or sodium sulphate solution in each dye vessel Stir the dye liquors and enter the wetted hanks at 40°C Turn the hanks frequently so as to obtain level dyeings. Slowly raise the temperature of dye-bath to 80°C withm 20 minutes Remove the hanks from the dye-bath, add second lot of 10 ml of sodium chloride solution or sodium sulphate solution in each dye vessel and stir well. Enter the hanks into the dye-bath and raise the temperature of the dye-bath to boil and continue the dyeing for 40 minutes. At the end of dyeing, squeeze the dyed hanks returning the squeezed out solution back to respective dye-baths and rinse them well in cold water Dry the hanks in oven at temperature not exceeding 70°C

A-6.4 The material shall be taken to have satisfied the requirement of the test, if the colour of the wool/cotton when dyed with the material is not different from the colour of the wool/cotton dyed with the approved/ standard dyestuff.

A-7. DETERMINATION OF VOLATILE MATTER

A-7.1 Procedure — Accurately weigh 2 to 3 g of the sample in a dried ground-glass petri dish with cover (about 5 cm diameter and 3 cm height) Uncover the dish and expose the material for three hours in an oven at 105°C Remove the dish, replace the cover, cool it in a desiccator and weigh Repeat the process till constant mass is obtained

A-7.2 Calculation

$$\text{Volatile matter, percent by mass} = \frac{M}{M_1} \times 100$$

where

M = loss in g of the material, and

M_1 = mass in g of the material taken for the test

A-8. DETERMINATION OF *p*H VALUE

A-8.1 Procedure — Accurately weigh 0 5 g of the dye and dissolve it in 100 ml of hot water Cool it to room temperature and determine the *p*H value by means of a suitable *p*H meter.

A-9. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-9.1 Procedure — Dissolve with stirring about 10 g of the accurately weighed material in about 500 ml of cold water Filter the solution through a weighed Gooch crucible and wash the residue repeatedly with cold water till the filterate is colourless Dry the Gooch crucible in an oven at 105°C for 3 hours Cool it in a desiccator and weigh Repeat the process till constant mass is obtained

A-9.2 Calculation

$$\text{Matter insoluble in water, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of the crucible and the residue, and

M_2 = mass in g of the material taken for the test.

A-10. DETERMINATION OF COLOUR VALUE OF AQUEOUS SOLUTION (0.002 5 PERCENT SOLUTION)

A-10.1 Procedure — Weigh accurately 0.250 g of the dye and dissolve it in hot water Transfer the solution into 1-litre graduated flask, giving the necessary water washings to the container till final water washing is free from the colour. Make up the volume with water to 1-litre m.ark. Shake it well

A-10.2 Pipette out 10 ml aliquot of the solution and transfer it quantitatively into 100-ml graduated flask as described in **A-10.1.** Make up the volume with water up to 1-litre mark Shake it well Take this prepared solution in a 1-cm cell and take the readings on Lovibond Tintometer in terms of yellow (*Y*), red (*R*), and blue (*B*).

A-11. TEST FOR SHADE (ON 0.5 PERCENT AQUEOUS SOLUTION)

A-11.1 Procedure

A-11.1.1 Weigh accurately 1.250 g of the dye Dissolve it in hot water Transfer the solution into 250-ml graduated flask giving necessary water washings to the container till final washing is free from the colour. Make up the volume to 250 ml with water. Shake it well.

A-11.1.2 Transfer the solution into 500-ml beaker. Take the shade of the solution by just dipping a Whatman filter paper piece (6 × 10 cm) into the solution. Dry the paper by hanging it at room temperature for about an hour

A-11.1.3 Similarly, prepare the solution of the approved/standard dye as in **A-11.1.1** and take the shade of the standard solution as in **A-11.1.2.**

A-11.2 The material shall be taken to have satisfied the requirement of the test if the shade of the dyed piece of the sample solution matches with the shade of the dyed piece of the standard solution.

NOTE — The shade te&t papers (as prepared in **A-11.1 2** and **A-11.1.3**) may further be examined by reflectance photometer at different wavelengths or by Lovibond Tintometer and may be compared

A-12. DETERMINATION OF CALCIUM OXIDE (CaO)

A-12.0 General — Two methods are prescribed, namely, (a) volumetric method, and (b) EDTA method

A-12.1 Volumetric Method

A-12.1.1 *Reagents*

A-12.1.1.1 *Dilute hydrochloric acid* — 1 20 and 1 2 (*v/v*).

A-12.1.1.2 *Ammonium chloride* — solid

A-12.1.1.3 *Ammonium nitrate* — solid

A-12.1.1.4 *Concentrated nitric acid* — *See* IS 264-1968*.

A-12.1.1.5 *Dilute ammonium hydroxide* — 1 1 (*v/v*)

A-12.1.1.6 *Ammonium hydroxide ammonium nitrate wash solution* — Add 5 ml of ammonium hydroxide solution (r. d 0.88) and 2 g of ammonium nitrate to 1 litre of water

A-12.1.1.7 *Ammonium oxalate solution* — saturated

A-12.1.1.8 *Dilute sulphuric acid* — 1 1 (*v/v*).

A-12.1.1.9 *Standard potassium permanganate solution* — 0.05 N

A-12.1.1.10 *Methyl red indicator* — Dissolve 0 1 g of methyl red in 100 ml of rectified spirit (*see* IS · 323-1959†)

A-12.1.2 *Procedure*

A-12.1.2.1 Weigh accurately 1 to 2 g of the dye, dried over sulphuric acid in a desiccator, in a platinum dish or crucible Heat over a bunsen burner raising the temperature slowly and keeping it red hot till the dye is completely ashed Cool the crucible and transfer the contents to a beaker Digest with 20 ml of hydrochloric acid (1 2), dilute with 50 ml of water and boil. Filter and wash the residue with dilute hot hydrochloric acid (1 : 20) Determine calcium in the filtrate

A-12.1.2.2 Take the solution in a 400-ml beaker, dilute to 200 ml, add 3 g of solid ammonium chloride and boil with 1 ml of concentrated nitric acid. Cool and neutralize with dilute ammonium hydroxide using 3 to 4 drops of methyl red indicator and then a slight excess of ammonia. Keep the solution on the water-bath for 10 to 12 minutes; filter through a Whatmam No 40 or **41** filter paper and wash with hot ammonium hydroxide-ammonium nitrate wash solution till the filtrate is free from chlorides.

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*Specification for nitric acid (*first revision*)
†Specification for rectified spint (*revised*)

A-12.1.2.3 Concentrate the filtrate to 200 ml and make the solution ammoniacal Bring to boil and add 10 to 15 ml of saturated ammonium oxalate solution. Continue boiling for 10 minutes and then place the beaker on a water-bath for about 2 hours, keeping the solution distinctly ammomacal Filter off the precipitate of calcium oxalate through a filter paper and wash with water at room temperature till the washings are free from oxalate

A-12.1.2.4 Transfer the precipitate to the beaker in which calcium was precipitated by punching the filter paper, wash down with 15 ml of dilute sulphuric acid and finally with 5 to 6 times with hot water through a jet to bring the calcium oxalate into solution. Dilute the contents of the beaker to about 200 ml. Heat to 60°C and titrate the solution while hot with standard potassium permanganate solution until a stable pink colour is obtained

A-12.1.3 *Calculation*

$$\text{Calcium oxide, percent by mass} = \frac{A \times B}{C} \times 2.803$$

where

A = volume in ml of standard permanganate solution consumed,
B = normality of the standard permanganate solution, and
C = mass in g of the sample taken

A-12-2 EDTA Method

A-12.2.1 *Beagents*

A-12.2.1.1 *Dilute hydrochloric acid* — 4 N approximately.

A-12.2.1.2 *Dilute ammonium hydroxide* — 15 percent (*m/m*).

A-12.2.1.3 *Methyl red indicator solution* — Dissolve 0 15 g in 500 ml of water

A-12.2.1.4 *Ammonium chloride ammonium hydroxide buffer solution* (*p*H 10) — Mix 350 ml of ammonium hydroxide (20 percent, *m/m*) with 54 g of ammonium chloride. Dilute with water and make up the volume to 1 000 ml

A-12.2.1.5 *Enochrome black T indicator* — Dissolve 0 1 g of eriochrome black T in 25 ml of methyl alcohol.

A-12.2.1.6 *Standard calcium solution* — 0.01 M. Weigh exactly 0 500 4 g of calcium carbonate and dissolve in minimum quantity of dilute hydrochloric acid Make up the volume with water to one litre in a volumetric flask

A-12.2.1.7 *Standard ethylene diamine tetraacetate* (*EDTA*) *solution* — Dissolve 3 72 g of disodium ethylene diamine tetraacetate dihydrate in water and make up the volume to 1 htre and standardize as follows:

Take 10 ml of standard calcium solution in a conical flask. Add 20 ml of water, 1 ml of eriochrome black T indicator and 2.5 ml of ammonium hydroxide-ammonium chloride buffer solution Heat to 40 to 50°C and then titrate with EDTA solution, maintaining the temperature between 40°C and 50°C until the colour changes to distinct blue

$$\text{Molarity of EDTA solution} = 10 \times \frac{M_1}{V_1}$$

where

M_1 = molarity of standard calcium solution, and

V_1 = volume m ml of EDTA solution used for titration

A-12.2.2 *Procedure* — Weigh accurately 1 g of the dye dried over sulphuric acid in a desiccator in a silica dish and heat it over the flame of a bunsen burner and then finally ignite in an electric furnace for 1 hour at about 500-600°C Dissolve the ash in 10 to 15 ml of dilute hydrochloric acid (do not boil), and neutralize with dilute ammonium hydroxide using methyl red indicator Heat over a water-bath for 10 to 15 minutes to digest any precipitate formed Filter the solution to remove any iron, aluminium, etc. Wash the residue with hot water to remove calcium chloride completely

A-12.2.2.1 Treat the filtrate and the washings with about 5 ml of ammonium hydroxide-ammonium chloride buffer solution and titrate with EDTA solution using eriochrome black T as indicator till blue end point is obtained

A-12.2.2.2 *Calculation* — Calculate the calcium oxide (CaO) content, percent by mass, on the basis that 1 ml of 0 01 molar EDTA solution is equivalent to 0 000 56 g of calcium oxide (CaO)

A-13. DETERMINATION OF SULPHATED ASH

A-13.1 Procedure — Ignite a 7-cm silica basin in a muffle furnace, cool in a desiccator and weigh Place about 2 g of the dye in the crucible and weigh accurately Heat the crucible on a low flame until the material is completely carbonized Burn off the carbonaceous matter by further heating in a muffle furnace. When the ash is practically white in colour, cool the crucible, add 5 drops of concentrated sulphuric acid (*see* IS · 266-1961*) and heat to fumes on a burner. When all the sulphuric acid has volatilized, cool the crucible in a desiccator and weigh accurately.

NOTE — The sample shall be heated cautiously to avoid undue swelling

* Specification for sulphuric acid (*revised*).

A-13.2 Calculation

$$\text{Sulphates ash. percent by mass} = \frac{100\,M}{M_1}$$

where

M = mass in g of the ignited ash, and

M_1 = mass m g of the material taken for the test.

A-14. TEST FOR PRESENCE OF STARCH, DEXTRIN AND GUM

A-14.0 Principle — Rectified spirit precipitates both starch and dextrin from aqueous solution of dyes while rectified spirit precipitates gum, starch and dextrin from acidic solution of dye.

A-14.1 Reagents

A-14.1.1 *Rectified Spirit* — 95 percent (*see* IS 323-1959*).

A-14.1.2 *Concentrated Hydrochloric Acid — See* IS 265-1962†

A-14.1.3 *Phloroglucinol* — powder

A-14.1.4 *Aniline*

A-14.1.5 *Iodine Solution* — approximately 0 1 *N*.

A-14.2 Procedure

A-14.2.1 *Detection of Gum, Starch and Dextrin* — Treat 10 ml of 2 percent dye solution with 20 ml of rectified spirit and after thorough shaking, dilute the solution with an equal volume of water Allow the solution undisturbed for 2 hours Formation of any precipitate or gelatinous matter indicates the presence of starchy materials and gums

A-14.2.2 *Distinction Between Gum and Dextrin* — Acidify the dye solution with concentrated hydrochloric acid and add enough rectified spirit Filter the precipitate, if any, wash with rectified spirit and treat with concentrated hydrochloric acid Divide the liquid into two parts:

a) to one part add, phloroglucinol. A rose violet colour indicates the presence of gum

b) to second part, boil and add aniline A rose red colour again shows the presence of gum

Negative reactions to above two tests show the absence of gum but presence of dextrin.

*Specification for rectified spint (*revised*).
†Specification for hydrochloric acid (*revised*)

A-14.2.3 *Distinction Between Dextrin and Starch* — Filter the solution containing the precipitate obtained in **A-14.2.1.** Take the precipitate in water, boil until the entire precipitate dissolves. To this solution add few drops of iodine solution. Brown colouration indicates dextrin, whereas blue colouration indicates starch.

APPENDIX B

(*Clause* 5 1)

SAMPLING OF DYES FOR WATKR-BASED WRITING INKS

B-1. GENERAL

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall be taken in a protected place

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 Samples shall be placed in clean, dry, airtight glass or other suitable containers on which the material has no action.

B-1.5 Each sample container after filling shall be sealed airtight with a stopper, and marked with full particulars of the material (see **4.2**).

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot If a consignment is declared or known to consist of different batches of manufacture, containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested from each lot for ascertaining the conformity of the material to the requirements of the specification.

B-2.2 The number (n) of containers to be chosen from the lot shall depend on the size of the lot and shall be in accordance with Table 4.

TABLE 4 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

LOT SIZE (*N*) (1)	No OF CONTAINERS TO BE CHOSEN (*n*) (2)
Up to 50	2
51 „ 100	3
101 „ 150	4
151 „ 300	5
101 „ 500	6
501 „ 800	7
801 „ 1 300	8
1 301 and above	9

B-2.3 These containers shall be chosen at random from the lot and an order to ensure the randomness of selection, procedure given in IS · 4905-1968* may be followed

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 Draw with an appropriate sampling instrument a small portion of material from different parts of each of the containers selected. The total quantity of the material so drawn from each container shall be sufficient for triplicate determination of all the characteristics given under 3.

B-3.1.2 Thoroughly mix all portions of material drawn from the same container. Out of these portions a small but equal quantity shall be taken from each selected container and shall be mixed up well together so as to form a composite sample The composite sample shall be divided into three equal parts, one for the purchaser another for the supplier and the third to be used as the referee sample

B-3.1.3 The remaining portions of the material from each container shall be divided into three equal parts These parts shall be transferred immediately to thoroughly dried bottles which shall then be sealed airtight with stoppers and labelled with all particulars of sampling given in **4.2**. The material in each such sealed bottle shall constitute test sample. These individual samples shall be separated into three identical sets of test samples in such a way that each set has a test sample representing each container selected One of these three sets shall be sent to the purchaser another to the supplier and third shall be used as referee sample

*Methods for random sampling.

B-3.2 Referee Sample — The referee sample consisting of a composite sample and a set of test sample marked for this purpose shall bear the seals of the purchaser and the supplier to be used in case of a dispute between the two

B-4. NUMBER OF TESTS

B-4.1 Tests for dye content, strength and colour value, shall be conducted individually on each of the samples constituting a set of test samples.

B-4.2 Tests for the remaining characteristics given in 3 shall be conducted on the composite sample

B-5. CRITERIA FOR CONFORMITY

B-5.1 Individual Samples — Test results for the dye content, strength and colour value shall be recorded.

B-5.1.1 *Dye Content and Strength* — The mean and range of test results for each of these characteristics shall be calculated as given below:

Mean ($\bar{X}$) = $\frac{\text{The sum of test results}}{\text{Number of tests}}$, and

Mean (R) = The difference between the maximum and the minimum values of the test results

The lot shall be considered to have satisfied and the requirements for the relevant characteristics if the expression $X \pm 0.6\ R$ lies within corresponding limits given in Table 3

B-5.1.2 *Colour Value* — The lot shall be considered to have met the requirements of colour value if all the test results on each of the individual samples satisfy the corresponding requirements given in Table 3.

B-5.2 Composite Sample — For the remaining characteristics prescribed under 3, the test results on the composite sample shall meet the corresponding requirements given in **3.1**, **3.2** and **3.3.**

B-5.3 The lot shall be declared as conforming to the requirements of this specification if **B-5.1** and **B-5.2** are satisfied, otherwise not.

INDIAN STANDARDS

ON

INKS AND ALLIED PRODUCTS

IS

219-1975	Ink powder and tablets (*second revision*)
220-1972	Ferro gallo tannate fountain pen ink (0 1 percent iron content) (*second rdvision*)
221-1977	Ink fluid blue black for permanent records (*second revision*)
222-1977	Ink fluid, Far general purposes (*second revision*)
393-1975	Ink, stamp pad (*second revision*)
394-1963	Ink, cloth marking (*revised*)
788-1971	Ink, drawing, waterproof, coloured (*first revision*)
789-1971	Ink, drawing, waterproof black (*first revision*)
1221 1971	Dye based fountain pen inks (*first revision*)
1222-1971	Ink, duplicating for twin ovlinder rotary machines (*second revision*)
1231-1957	Ink stencil, oil base, for marking porous surfaces, colour as required
1333-1973	Ink, duplicating for single drum rotary machines (*first revision*)
1379-1959	Ink stencil, oil base for marking non porous surfaces, colour as required
1380-1959	Ink, finger printing, black
1440-1959	Ink, metal stamp, black
1551-1976	Carbon papers, for typewriters (*first revision*)
1581-1971	Ferro gallo tannate fountain pen ink (0 2 percent iron content) (*first revision*)
2210-1962	Dye, methylene blue, for ink industry
2247-1962	Dye, ink blue, for ink industry
2694-1963	School chalks, moulded, white
3450-1976	Carbon papers, handwriting (*first revision*)
4174-1977	Typewriter ribbons, cotton (*first revision*)
1175-1967	Correcting fluid
4222-1967	Coloured chalks, moulded
4395-1967	Glossary of terms relating to inks and allied industries
4717-1977	Pads foi rubber stamps (*first revision*)
5086-1969	Stencil paper
5805-1970	Ball point pen ink
6897-1973	Methods of test for tannic acid
6898-1073	Methods of test for gallic acid
8075-1976	Back coated carbon papers for typewriter
8100-1976	Water colours for students
8101 1976	Poster colours
8277-1976	Water based records inks
8279-1976	Hectographic carbon paper
8642-1977	Dyes for water-based writing inks
8643-1977	Dye, methyl violet, for stamp-pad ink

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